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The apparatus, known commercially as Robot Met. 3D, permits full three-dimensional characterization of materials of interest to the Department of Defense. Several projects are currently benefitting from the availability of this instrument. One project (DARP) through Northrop-Grumman) is focused on fatigue crack initiation in 7075 wing skin materials: a second project (AFOSR-STW21) deals with grain size control in Ni-based with super alloys for gas turbine applications.

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AFOSR-DURIP Award FA9550-07-1-0287
Acquisition of Robotized Metallographic Equipment with Automated Laue Pattern Analysis
Final Project Performance Report

M. De Graef

A.D. Rollett

Principal Investigators

October 23, 2008

1 Project Summary

This project (AFOSR-DURIP Award FA9550-07-1-0287) received funds ($ 672,414) for the acquisition of components of a system designed to automatically acquire serial sectioning images of microstructures in advanced engineering materials, along with selected orientational information.

The apparatus, known commercially as *RoboMet.3D*, permits full three-dimensional characterization of materials of interest to the Department of Defense. Several projects are currently benefitting from the availability of this instrument: One project (DARPA through Northrop-Grumman) is focused on fatigue crack initiation in 7075 wing skin materials; a second project (AFOSR-STW21) deals with grain size control in Ni-based superalloys for gas turbine applications, and the third program (ONR, D3D) deals with the creation of experimental and computational tools for the digital representation of microstructures, predominantly applied to two-phase titanium alloys. The DURIP grant has also had a significant impact on the education of engineers in the Department of Materials Science and Engineering at CMU, exposing both graduate and undergraduate students to problems and techniques of interest to DoD.

In addition to the standard RobotMet.3D instrument, we have also acquired components for the creation of an automated Laue diffractometer station, which is currently (October-November 2008) being added to the RoboMet.3D system. This diffractometer will allow for the automated acquisition of orientational data in materials with a grain size of several tens of microns.

Finally, we have installed an active stereo projection system in the Materials Characterization Laboratory in Roberts Engineering Hall; this system allows for 3-D visualization of data sets acquired with the RoboMet.3D instrument.

2 Acquired Equipment List

The following lists the major components of the RoboMet.3D instrument and associated sub-systems along with the total cost:

- RoboMet.3D automated metallography instrument .... $ 248,800.00
- Carl Zeiss AxioObserver inverted optical microscope .... $ 74,185.50
- 3D active stereo visualization system .... $ 52,209.37
- Preconfigured Photonic Science dual lens coupled Gemstar-2 intensified x-ray Laue imaging camera .... $ 86,200.00
- Nikon flat top stage for Laue diffractometer .... $ 15,313.23
- Multiprep precision polishing system .... $ 20,577.86
- Microfocus x-ray source with focusing optics and associated metrology .... $ 169,331.00
- Machine shop charges .... $ 2,002.75

- **Total Project Cost** .... $ 668,619,71

3 Project Narrative

The RoboMet.3D instrument was ordered from UES, Inc., in August of 2007, and installed in the laboratory of P.I. De Graef in March of 2008. The instrument consists of a robot arm, an automated polisher station, a chemical etching, rinsing and drying station, and an optical microscope with high precision x-y-stage. A second polisher station was ordered and placed outside of the RoboMet.3D enclosure to facilitate experimenting with different polishing conditions while a sample run is ongoing inside the enclosure. The overall setup is shown in the photograph below.
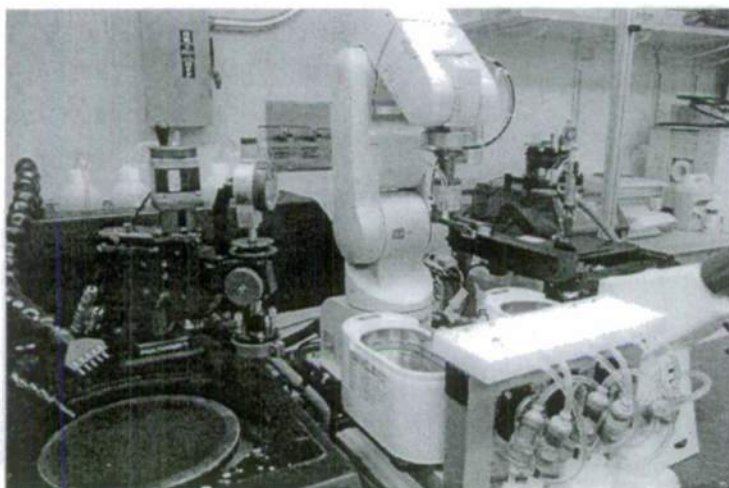
The PIs and six graduate students received extensive training in the use of the robot arm and the overall use of the instrument. In addition, several students visited the Ohio State Campus in Columbus, OH, during the Summer of 2008 to receive additional RoboMet.3D training (a similar instrument is located in the Department of Materials Science and Engineering at OSU). An undergraduate project on porous titanium for bio applications recently started to make use of RoboMet.3D instrument.

After the installation of the instrument was completed, the final design for the x-ray Laue diffractometer system could begin. Components for this system, which was designed in collaboration with Dr. Daniel Brau of Photonic Science Ltd., were ordered in June and July of 2008.[1] The Laue system, which will be fully operational on November 1, 2008, will be capable of producing Laue diffraction patterns using a beam spot size of about 10 μm diameter, resulting in an acquisition time

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[1]The PIs requested and received a three-month no-cost extension to the DURIP project, during which the final components of the system were ordered.

of 10-30 seconds per pattern. This will enable us to acquire orientational information in addition to the visual microstructural information that can be extracted from the optical micrographs.

To support the acquisition and reconstruction of 3-D microstructural data, we have installed an active stereo projection system in the Materials Characterization Laboratory in the Department of Materials Science and Engineering (one floor down from the RoboMet.3D instrument). This stereo system allows for the visualization of complex 3-D microstructures by means of an infra-red synchronized shuttering system and two digital projectors. This system has been operational since January of 2008.

The RoboMet.3D system is in active use at the moment to characterize the microstructure of a sample of Ti-6-4 in order to determine its 3-D microstructure. This work is in support of the ONR/DARPA Digital 3-D Structures program, which is executed as a subcontract to the Ohio State University (P.I. H. Fraser). After optimization of the chemical etching procedure, we are now acquiring serial section data using long term runs (multiple days of continued uninterrupted operation).

Another project is to develop the polishing and etching techniques that will allow us to characterize the (often complex) 3D fatigue crack paths in AA7075 and AA7050. Since these alloys have significant (1 to 2 %) volume fractions of coarse intermetallic particles, the interaction of fatigue cracks with both the grain structure (orientations) and with the particle structure is crucial fro understanding the behavior of short cracks (less than about 250 microns). In the early stages of the effort, the surfaces will be intermittently scanned with EBSD (i.e., every few layers) in order to associate orientations with the grain structure. As the Laue system becomes integrated with the RoboMet.3D instrument, EBSD observations will be initially used to verify that the X-ray system is working correctly, and eventually only the x-ray system will be used.